

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Kambe et al.
Serial No.: 08/962,362
Filed : October 31, 1997
For : PHOSPHORS
Docket No.: N19.12-0006

Group Art Unit:
2879
Examiner: M. Day

DECLARATION UNDER 37 C.F.R. § 1.132

Express Mail: EL418983858US
Date of Deposit: March 24, 2000

Assistant Commissioner for Patents
Washington, D.C. 20231

I, Rajiv K. Singh, Ph.D., hereby declare as follows:

1. I am presently a Professor of Material Science and Engineering at the University of Florida at Gainesville. Also, I am also Director of the Characterization, Research Instrumentation and Testbed Facility of the Engineering Research Center for Particle Science and Technology at the University of Florida. Apart from my academic responsibilities, I provide consulting services through R. K. Singh Consulting Inc.
2. I received my Ph.D. degree in 1989 in Material Science and Engineering from North Carolina State University, Raleigh, NC.
3. I have been on the faculty at the University of Florida since 1990. I was promoted to Associate Professor with tenure in 1995 and to full Professor in 1997. A copy of my Curriculum Vitae is attached.
4. My recent accomplishments include receiving a National Science Foundation Young Investigator Award in 1994 and the Hardy Gold Metal for Outstanding Contributions in Material Science in 1995. I was a Distinguished Visiting Professor/Scientist at National University of Singapore (1999) and National Institute for Materials and Chemical Research, Tsukuba, Japan (2000). I am the

author or co-author of more than 293 scientific articles and conference proceedings. I have co-edited five books and guest edited five journal issues.

5. I am under a Consulting Agreement with NanoGram Corporation to provide consulting services in the area of chemical-mechanical planarization. I am not a shareholder in NanoGram Corporation.

6. I have read carefully the pending claims of the above noted patent application entitled "PHOSPHORS" and U.S. Patent 5,442,254 to Jaskie (the Jaskie patent). I did not participate in any capacity with the preparation of the PHOSPHORS patent application.

7. I am very familiar with approaches that have been attempted for separating nanoparticles by filtration. To my knowledge, no experimental results based on the separation of nanoparticles by the wet filtration approaches described in the Jaskie patent at column 7, lines 28-40 have ever been reported in the public literature. Since such chromatography techniques are not known for the separation of nanoparticles, a person of skill in the art would expect to expend a substantial amount of inventive effort to attempt to practice the wet filtration techniques. Without at least some preliminary results to support the basic principles underlying the effort, there would be no reasonable expectation of eventual success at applying the wet filtration approach. In my opinion, the wet filtration approaches described in the Jaskie patent at column 7, lines 28-40 are highly speculative, and the description in the Jaskie patent does not provide a reasonable expectation of successfully separating a collection of nanoparticles to isolate a particle population with a desired narrow range of particle sizes.

8. Since the Jaskie wet filtration techniques would be difficult or impossible to scale up to commercial quantities, it is unlikely that any effort will ever be spent on developing such approaches. The only possibility for the expenditure of experimental effort on such wet filtration techniques would be to

satisfy academic curiosity. In the biological sciences, gel electrophoresis is an important technique for the characterization of biochemical preparations. However, there are other long established approaches for characterizing nanoparticles.

9. I am aware of considerable amounts of effort expended using conventional size exclusion filtration for the preparation of nanoparticle collections. At the time of filing the PHOSPHORS patent application on October 31, 1997, there were no filtration approaches publicly known that could create a collection of phosphorescent nanoparticles with a very narrow size distribution as disclosed and claimed in the PHOSPHORS patent application. At best, these filtration techniques could only exclude micron scale contaminants.

10. Based on my extensive knowledge in the nanoparticle field, it is my opinion that the "tuning (size selection)" described in the Jaskie patent at column 7, lines 28-30 could not be accomplished based on publicly available filtration methods as of October 31, 1997. Specifically, tuning could not be performed by the wet filtration approach outlined in the Jaskie patent without the expenditure of an undue amount of experimentation, if at all.

11. I declare that all statements made herein that are of my own knowledge are true and that all statements that are made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Date:

March 21 '00

By:

Rajiv K. Singh

Rajiv K. Singh, Ph.D.



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Research Interests:

Innovative processing of materials; Laser processing; thin films; transient thermal phenomena; superconducting and dielectric (low K and high K) thin films; diamond and related materials, rapid thermal processing of elemental and wide band gap semiconductors, chemical-mechanical planarization, particulate coatings; semiconductor processing; modeling of transient thermal processing; flat panel displays, Angstrom scale advanced materials characterization, oxide thin films & electronics, gallium nitride and diamond crystal growth, nanoparticle synthesis and processing, front and back end semiconductor cleaning, phosphors and flat panel displays, thin film batteries.

Education

Ph.D. Materials Science and Engineering, North Carolina State University, Raleigh, 1989
M.S. Materials Science and Engineering, North Carolina State University, Raleigh, 1987
B.S. Chemical Engineering, Jadavpur University, Calcutta, India, 1985

Positions Held

97-pre **Professor**, Materials Science and Engr., University of Florida
94-pre **Director**, Characterization Research Instrumentation and Testbed (CRIT) Facility,
Engineering Research Center (ERC), University of Florida
96-pre **Thrust Leader**, Chemical Mechanical Planarization (CMP), ERC Univ of Florida
94-pre **Thrust Leader**, Engineered Particulates, ERC, Univ. Florida
95-97 **Associate Professor**, Materials Science and Engr., University of Florida
90- 94 **Assistant Professor**, University of Florida, Gainesville, FL

Awards/Honors

2000 - **Distinguished Visiting Scientist**, NIRIM, Tsukuba, Japan
1999 **Distinguished Visiting Professor**, National University of Singapore, Singapore
1998 **Distinguished Visiting Professor**, University of Osaka, Osaka, Japan
1995 **Hardy Gold Medal** from TMS/AIME for Outstanding Contributions in Materials Science
1994 **NSF Young Investigator Award**
94-97 **Visiting Fellow**, Center for Ultrafast Optical Science (CUOS), University of Michigan
1993 **IEEE Senior Member Award**
1991 **IBM Faculty Development Award**
1989 **MRS Best Graduate Student Award**
1985 **Alumni Gold Medal** for Best Overall Graduating Senior from the University
1985 **Laha Silver Medal** for Best Graduate from College of Engineering

Publications:

Over 293 papers (> 268 published/in print & 25 submitted for various materials science and engineering journals (*Science*, *Physical Review B*, *Applied Physics Letters*, *Journal of Materials Research*, *Materials Science and Engineering B*, etc.) and Conference Proceedings. Published over 32

original, *principal author papers in* App. Phy. Lett. (The most cited electronic materials/applied physics based journal), and 7 papers published in Physical Review B

Invited and Contributed Talks

Presented more than **110** invited talks at international conferences (MRS, SPIE, TMS, APS, ASME, etc.) and academic and research institutions (MIT, Columbia, Purdue, ORNL, Westinghouse, etc.). Also group presented over **250** technical papers at international conferences

Books and Guest Editorships (Edited 5 books & Guest Editor of 5 Journal Issues)

- (1) R. K. Singh, D. Norton, J Cheung and J. Narayan and L.D. Laude , *Eds "Laser Processing of Materials: Fundamentals and Advanced Applications*, MRS Proceedings Vol 397, Pittsburgh, PA, 1996
- (2). N. M. Ravindra and R.K. Singh, "*Transient Thermal Processing of Materials*", TMS, Warrendale April. 1996
- (3). K. Gonsalves, M. Baraton, J.. Chen, J. Akkara, R. K. Singh and H. Hofmann , "*Surface Controlled Nanoscale and Microscale Materials for High Value Added Applications*, MRS Proceedings Vol 501 , Pittsburgh, PA, March 1998
- (4). R.K. Singh, D. Lowdnes, J. Narayan, D. Chrisey , T. Kawai, and E. Fogarassy, Editors, *Advances in Laser Ablation of Materials*", MRS Proceedings for Spring 1998 .
- (5). R. K. Singh and D. Kumar, "*Advances in Pulsed Laser Deposition of Thin Films*", Kluwer publishers, (1998)
- (1) *Guest Editor* of September 1994, Vol 23 issue of *Journal of Electronic Materials* titled " *Novel Issues in Photonic Materials*"
- (2) *Guest Editor* of Jan,96, Vol 1 issue of *Journal of Electronic Materials* titled "*Ion and Laser Beam Processing of Electronic Materials*"
- (3) *Guest Editor* of Materials Science and Engr. B, on *Laser Processing of Electronic Materials*, Jan 1997
- (4) *Guest Editor* of November 1997 Issue of *Journal of Electronic Materials* on " *Low Energy Beam Processing of materials.*"
- (5) *Guest Editor* of September 1998 Issue of *Journal of Electronic Materials* on " *Chemical-Mechanical Polishing of Semiconductors.*

Teaching Accomplishments

Developed four new courses: "Beam-Solid Interactions", "Thin Films" & "Math. Methods", "Survey of Materials Analysis" in the graduate MS&E program
 Graduated 12 Master's and 10 Ph.D Students; Presently thesis advisor to 9 Ph. D Students
 8 students awarded best paper/fellowships for their undergraduate/graduate research projects.
 Developing CD-ROM materials and multi-media classroom for the NSF ERC project.
 Established ParTiN (Particle technology) Hypertext Network for educational & ERC programs on the WWW (World Wide Web)

Corporate Interactions

Direct Research Interactions with several companies including IBM, Intel, Motorola, Ashland Chemical, Westinghouse, Lucent Technology, Applied Materials, Sony, Glaxo Wellcome, Lockheed Martin, Astra Zeneca, Purdue Pharma
 Licensing Discussions with Sony, Nara Machinery, Astra Zeneca, Glaxo, etc.
 Corporate funding over 200 K/yr .

Copyrighted Softwares (3 copyrighted softwares) including

(A) **SLIM** (*Simulation of Laser Interaction with Materials*, 36,000 coded lines, 1992) software.

This first of its kind software is being used by more than 50 R&D groups (IBM, LANL, ORNL, etc.) in the world. This software calculates the transient thermal induced laser effects like melting, crystallization and ablation of materials. This software has had sales greater than \$ 60 K worldwide in the last four years. Two new versions (one based on DOS C++ and the other on Windows platform) have been developed..

Patents (from a total of 30 disclosures:[14 patents, 10 awarded/pending(final stage) and 4 filed])

(1) **High Surface Area Metals and Ceramics** [US Patent 5,473,138] . A unique laser technique has been developed to increase the surface areas of ceramics, metals and composites. This technique involves the use of multiple-pulse laser irradiation under controlled energy window conditions.

(2) **Enhanced Chemical Vapor Deposition of Diamond** [US Patent 5,485,804 {1996}, Filed for worldwide patent} Novel colloidal method for large area nucleation, of diamond. *This method has been used to make the world's largest single monolithic piece of diamond which has a diameter greater than 11" and weighs over 1600 carats.*

Conference Chairs [Organized 16 international conferences on innovative processing and characterization of materials]

- (1) Chair of Symposium. on "Beam Processing of Materials", **TMS/AIME Winter meeting**, Chicago Nov 92;
- (2) Co-Chairman of Symposium on "Innovative Processing of Electronic and Photonic Materials" **TMS/AIME Annual Meeting**, Denver, Feb 1993;
- (3) Chairman of Conference on "Advanced Laser Processing of Materials" **Engineering Foundation** Conf., Palm Coast, FL, May 1-6 1994;
- (4) Chair of Symposium on " Ion Beam Processing of Materials" **TMS Spring Meeting**, Las Vegas, Feb 1995
- (5) Co-Chair of symposium on "Laser Processing of Materials" **American Physical Society**, San Diego, March 1995
- (6) Chair of Symposium on " Advanced Laser Processing of Materials: Fundamentals and Advanced Applications" **MRS Meeting**, Boston Nov 1995
- (7) Co-Chair, Symposia on "Transient Thermal Processing of Materials", **TMS Annual Meeting**, Anaheim, CA Feb, 1996
- (8) Chairman of symposium on, " Low Energy Beam Processes", **TMS Annual Meeting**, Orlando , FL Feb, 1997
- (9) Chairman of symposium on " Particulate Coatings", **MRS Fall Meeting**, Boston November, 1997
- (10) Co-Chairman of " Laser and Ion Beam Processing of Materials", **International Union of Materials Research Societies (IUMRS)**, Chiba, Japan, September 1997
- (11) Co-Chairman," Transient Thermal Processing of Materials , **TMS Annual Meeting**, San Antonio, Feb 1998
- (12) Co-Chairman," Chemical Mechanical Planarization of Materials Symposia, **TMS Annual Meeting**, San Antonio, Feb 1998
- (13) Chairman, " Advances in Pulsed Laser Ablation of Materials", **MRS Spring Meeting**, San Francisco, April, 1998

- (14) Co-Chairman, "Particulate Coatings" **5th World Congress on Particle Science and Technology**, Brighton UK, July 1998
- (15) Co-Chairman "Rapid Thermal Processing of Materials"- **European MRS Meeting**, Strasbourg, June, 1998
- (16) Chairman, "Chemical Mechanical Polishing Symposia", **MRS Spring Meeting**, San Francisco, April 2000

Invited Review Articles

- 1. "Pulsed Laser Deposition of Thin Films", *Materials Science and Reports* in March, (1998)
- 2. "SLIM, A Personal Computer Based Simulation of Laser Interaction With Materials", *J Journal of Materials*, **44**, 20 (1992)
- 3. "Pulsed Laser Deposition and Processing of Superconducting Thin Films", *J. of Materials* **43**, 13 (1991)

Book Chapters

- 1. D. Gilbert and R. K. Singh, "Boron Nitride Interfaces", in " *Properties of Group III Nitrides*, Edited by James Edgar, *Imspec* publication, London 1995
- 2. R. K. Singh, "Raman Based Optical Properties of YBaCuO Surfaces", in " *Optical Properties of Materials*", Eds R. Hummel, CRC Press, 1996
- 3. R.K. Singh and D. Kumar, "Pulsed Laser Deposition of Superconducting Thin Films", *Materials Science and Engr. Reports* (in press, 1996)
- 4. R. K. Singh and D. Kumar, "Thermal Annealing of Semiconductors", *Encyclopedia of Applied Physics*, VCH Publishers (1996)
- 5. D. Gilbert and R. K. Singh, "Diamond Deposition for Electronic Applications", Eds . S. Pearton on *Wide Band Gap Semiconductors*", VCH Publishers 1997

Reviews

Reviewer for NSF, DOE, Physical Review B, Materials Science and Engineering, Journal of Applied Physics, Applied Physics Letter, Journal of Materials Research and Physica C.
Invited to serve as panel members for several NSF initiatives

Memberships and Committee Chairmanships:

Member of MRS, ASM, TMS, IEEE (senior member), APS, AIChE, SPIE
Chairman: Thin Films and Interfaces Committee, TMS (1993 -1996)
Member: Laser Processing of Materials Committee, CLEO

Institutional Impact

- (A) Helped establish the 60 million, NSF Funded **Engineering Research Center (ERC)** on particle science and technology at the University of Florida:
- (B) Leader of team for the NSF MRSEC Proposal submitted in 1995 & 1997
- (C) Established cross-disciplinary multi P.I programs in Rapid Thermal Processing, Particle Coating Technology, Radiation Damage in Electronic Devices, and Beam Processing of Materials:
- (D) Established industrial and scientific collaborations with various institutions:
- (E) Established international collaborations with

(i) CNRS, Strasbourg France, (ii) University of Melbourne, Australia, (iii) University of Osaka, Japan, (iv) Keio University, Japan, (v) EPFL, Lausanne, Switzerland, (vi) Unicamp, Sao Paulo Brazil, (vii) National University, Singapore